ASSESMENT OF SPONTANEOUS HEATING SUSCEPTIBILITY OF COALS USING DIFFERENTIAL THERMAL ANALYSIS

A THESIS SUBMITTED IN PARTIAL FULFILLMENT
OF THE REQUIREMENTS FOR THE DEGREE OF

BACHELOR OF TECHNOLOGY

IN

MINING ENGINEERING

BY

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DEPARTMENT OF MINING ENGINEERING

NATIONAL INSTITUTE OF TECHNOLOGY

ROURKELA - 769008

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CERTIFICATE

This is to certify that the thesis entitled **"ASSESMENT OF SPONTANEOUS HEATING SUSCEPTIBILITY OF COALS USING DIFFERENTIAL THERMAL ANALYSIS"** submitted by **Sri Abhishek Jain** in partial fulfillment of the requirements for the award of Bachelor of Technology degree in Mining Engineering at the National Institute of Technology, Rourkela is an authentic work carried out by him under my supervision and guidance.

To the best of my knowledge, the matter embodied in the thesis has not been submitted to any other University/Institute for the award of any Degree or Diploma.

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CONTENTS

ABSTRACT

The auto-oxidation of coal is a complex physico-chemical process which is accompanied by the absorption of oxygen, formation of coal oxygen complexes and their decomposition leading to the liberation of heat. This complexity of the process is enormous because of the great diversity in the coal substance with the associated mineral matter and the condition of the heterogeneous mass, concurrent and over lapping reactions takes place which are very difficult to separate out. The rate of oxidation of coal at ambient temperature gives a measure of the proneness of coal to auto oxidation. From the economics stand point, even a small incidence in auto oxidation causes loss of production when it becomes necessary to seal off a section or a district resulting in the possible loss of machinery and the deterioration of reserves is potentially great. Fires in coal mines are one of the serious problems of Indian coal mining industry. In India every year many incidence of the fire occur in the underground and surface due to spontaneous combustion. Mostly the fire occurring due to spontaneous combustion per year is a function of the quality of the coal and the circumstances which it is subjected. They endanger not only the valuable lives of men in mine but also cause considerable economic losses to the organization affected by them. History of coal mines fires can be traced back to the year 1865, when the first fire was reported in Raniganj coal fields. Over 140 years fires have been reported till the year1967 from both Jharia and Raniganj coal fields and superior quality non-cocking coal in Raniganj coal fields.

Various methods have been used in laboratory for the study of relative tendency of coals to heat spontaneously. Most of the methods are based on the measurement of the oxidation rate and ignition temperature. The propensity of different coal to self heating has been studied in the laboratory using crossing point temperature, different thermal analysis, oxygen absorption method etc. the result of the test analyzed according to readily recognizable characteristics temperature and rates of heating under non adiabatic condition. These simple indices have historically been considered to be prediction of self heating liability.

: List of figures:

: List of tables:

Chapter 1

INTRODUCTION

GENERAL

CONCEPT OF SPONTANEOUS HEATING

HISTORY OF SPONTANEOUS COMBUSTION

1. I N T R O D U C T I O N

1.1 GENERAL

The auto-oxidation of coal is a complex physico-chemical process which is accompanied by the absorption of oxygen, formation of coal oxygen complexes and their decomposition leading to the liberation of heat. This complexity of the process is enormous because of the great diversity in the coal substance with the associated mineral matter and the condition of the heterogeneous mass, concurrent and over lapping reactions takes place which are very difficult to separate out. The rate of oxidation of coal at ambient temperature gives a measure of the proneness of coal to auto oxidation. From the economics stand point, even a small incidence in auto oxidation causes loss of production when it becomes necessary to seal off a section or a district resulting in the possible loss of machinery and the deterioration of reserves is potentially great.

1.2 CONCEPT OF SPONTANEOUS HEATING

Spontaneous combustion of coal is the process of self heating resulting eventually in its ignition without the application of external heat. When coal is exposed to air it absorbs oxygen at the exposed surface. Some fraction of the exposed coal substance absorbs oxygen at a taster rate than others and the oxidation results in the formation of gases. Mainly CO, CO_2, water vapor along with the evolution of heat during the chemical reaction. The process takes place at normal temperature, but it is slow and the heat evolved is not carried away by air. If the rate of dissipation of heat is slow compared with the evolution of heat by oxidation. There is gradually build up of heat and rise temperature reaches the ignition point of coal with then catches fire.

The ignition temperature of bituminous coal is nearly 160-170 ^{0}C and of anthracite coal nearly 185^{0}C. A good air current will effectively prevent oxidation. Once the coal reaches it ignition point, the air supply to it will only increase the combustion. The term spontaneous combustion express a relative value which are classified as highly susceptible or not.

1.3 HISTORY OF SPONTANEOUS COMBUTION

Fires in coal mines are one of the serious problems of Indian coal mining industry. In India every year many incidence of the fire occur in the under ground and surface due to

spontaneous combustion. Mostly the fire occurring due to spontaneous combustion per year is a function of the quality of the coal and the circumstances which it is subjected.

The problem of this type of fire has been existing in India for several years. Analysis of the causes of the coal mines fire reveal that they start either from an open fire over the external mining agencies or originate due to varying nature of coal. Fires occur whenever and wherever combustible material is present in mine working. They endanger not only the valuable lives of men in mine but also cause considerable economic losses to the organization affected by them. History of coal mines fires can be traced back to the year 1865, when the first fire was reported in Raniganj Coelfields. Over 140 years fires have been reported till the year1967 from both Jharia and Raniganj coal fields and superior quality non-cocking coal in Raniganj coal fields. These fires not only continue to spread to adjoin areas, adding to the losses but also prevent economic exploitation of the seam in the vicinity. Again the open fire in these fields causes environmental pollution by emission of huge quantities of steam, smoke and noxious gases posing a serious health hazards.

In Indian coal mines 80% of the coal fires occur due to spontaneous combustion. The main aspect of starting the fire in India is that the coal seams are thicker and there is a tendency of spontaneous heating during the depillaring operation. The problem of extraction of thick seam and coal standing in pillars is a serious one particularly in cases where they are with high moisture, high volatile and low ash content which are more liable to spontaneous combustion. It is not practicable to extract all the coal by caving method or even by complete packing under Indian mining condition,. Pillars standing for long time are liable to deteriorate in straight and spilling may occur. If coal is heated to about 93 ^{0}C and O_2 in the air is available, the oxidation will take place at accelerated rate and if the heat of decapitation is less than heat of accumulation then coal bed temperature increases, leading to fire.

Chapter 2

LITERATURE REVIEW

FACTORS AFFECTING SPPONTANEOUS HEATING OF COAL

THEORIES OF SPONTANEOUS COMBUSTION

2. FACTORS AFFECTING SPONTANEOUS HEATING OF COAL

Spontaneous combustion is one of the major hazards of mining from both the safety and economics aspect. around 80 years ago Hall Dane stated that, 'A mining engineer has to consider many other things than a mere prevention of the spontaneous fires .In particular, he has to work a mine so that it shall pay, and that the method of working shall be as safe as possible all round . He may thus have to coal underground at the risk of spontaneous fires, or in the other ways to adopt methods of working which extends the risk of heating.

However, it is the contention that competent mining engineer will design the mine to minimize all risks to the safety of the personnel and property and to maximize profitability. Every incident, however small, if not dealt with effectively and efficiently in the early stages can develop into open fires or into an explosion of gas or dust, with devastating results.

In term spontaneous combustibility may be used to express a value by which coal may be termed highly combustible or poorly combustible .Spontaneous heating occurs only in certain seams and these susceptible seams are frequently adjacent to seams worked by the same method and subjected to the same tectonics, but which have never been the site of combustion. A few seams have only been susceptible in one area and, again, some seams are more susceptible than others. In this chapter the effect of all the factors on spontaneity of coals are discussed.

The whole phenomenon of the spontaneous combustion as aggregate effects of the situation which may be classified as:-

A-**Seam factors**:

1-rank	7-sulphur
2-petrographic composition	8-the effect of previous oxidation
3-temperature	9-physical properties
4-available air	10-heating due to earth movement
5-particle size	11-bacteria
6-moisture	12-other minerals

B-Geological factors:

1-seam thickness
2-seam gradient
3-caving characteristics
4-faulting
5- coal outbursts
6- friability
7- depth of cover

C-Mining Factors:

1-mining methods
2-rate of advance
3-pillar conditions
4-roof condition
5-crushing
6-packing
7-effect of timber
8-leakage
9-multi-seam working
10-Coal losses
11-main roads
12- worked out areas
13-heat from machines
14-stowing
15-ventilating pressure
16-change in humidity

2.1 SEAM FACTORS

2.1.1 Rank: The rank of the coal depends on the character of the original plant debris from which it was formed and the amount of change that its organic matter has undergone during the period of formation. An increasing carbon content and with its decreasing oxygen content is the most commonly accepted criteria of increasing rank. The higher the rank, e.g. anthracite, the slower the oxidation rate, whilst lignite of the low rank, oxidizes so rapidly that it is often stated that is can't be stored after mining without ignition.

There are however numerous anomalies to a straight rank order. One part of a seam may be particularly liable to spontaneous heating, a seam of higher rank may prove more troublesome than one of lower rank or even the same in different mines may react differently.

2.1.2. Petrographic composition: The National coal board carried out a series of oxidation tests on hand picked petrographic constituents from five coals ranging from high ranking coking coal to low rank bituminous coal. The result of this test showed that in all cases fusain was the least reactive, and in general, durain was more reactive than vitrain. These results enabled calculation to be made of the reaction velocities of vitrinite, exinite and inertinite and showed that above 165 degree Fahrenheit exinite has a much greater oxidation rate than the other two constituents. Thus it appears that a count of these macerals may be

useful, together with rank in determining the susceptibility of coals to spontaneous combustion.

2.1.3. Temperature: The absorption of oxygen is more rapid as the temperature increases. There is a pronounced temperature coefficient of oxidation and the average rate of oxidation approximately doubles for every rise of 18 degree Fahrenheit.

2.1.4. Available air: Where there is a small amount of air the rate of oxidation is very slow and there is no appreciable rise in temperature .Where there are large quantities of air passing over or through the coal, any heat produced will invariably be carried away so that the temperature does not rise and the oxidation rate remains at a low level. However, between these two limits there is a state when the air quantity is sufficiently to promote oxidation but not sufficient to carry away the heat formed, so that there is an accelerating rate of oxidation until ignition occurs.

2.1.5. Particle size: A solid coal face generally presents very little danger of spontaneous combustion, partly due to the small surface area and partly due to the very low permeability of solid coal gases. It is however, generally when coal is shattered in mining, or broken by roof pressure, when falls and faulting occur that spontaneous combustion is likely to take place. It is the small coal that is mainly responsible for the heating. The air passes into the mass oxidizes a little of the coal near the outer surface. They produces a slight rise in temperature, so that as the air penetrates deeper and deeper, it becomes warmer and warmer and although part of its oxygen has been absorbed there is still enough to produce oxidation. Consequently, it is at some distance inside the mass that heating develops most rapidly. It should be noted that a flame is due to the combustion of gas and this requires that a moderately high proportion of oxygen be present. Once the oxidation process has gone beyond the early stages and heat is accumulating, it is only a matter of time before actual ignition takes place.

2.1.6. Moisture: The effect of moisture on spontaneous heating is uncertain. A small quantity seems to assists rather than retard the heating whilst large quantities of moisture retard the heating. However, as in a surface stockpile, alternate drying and wetting of the coal accelerates the heating process.

2.1.7. Sulphur: From the first publication on spontaneous combustion, R. Plott in 1686, until about the middle of the nineteenth century it was assumed that sulphur in the form of pyrites was the main cause of spontaneous combustion. However, it was shown that coal even in the absence of sulphides would absorb oxygen and heat spontaneously. However,

further research work modified this view and led to the present theory that pyrites plays a subsidiary role in promoting deterioration and spontaneous combustion.

2.1.8. Other minerals: Many other chemicals affect the rate of oxidation to some extent, either accelerating or retarding it. Alkalis can act as accelerators, and borates and calcium chloride as retardant.

From the effects of previous oxidation from experimental work on the effects of preheating coals in vacuum, cooling ,and then comparing their oxygen reaction with that of untreated coals, the following conclusion were drawn :

- By preheating coals their liability to spontaneous combustion is greatly increased.
- Lump coal previously almost impervious to air and thus without danger from the point of view of heating , may become ,though being in the neighborhood of a fire, a source of danger due to a large increase in its oxygen capacity.
- Coal in a sealed area, even after a fire has become completely extinguished, may also have formed fine coal caused by reduction in the strength, following partial distillation..

2.1.9. Bacteria: Two papers during this period deserve comment. The first reference, stated that spontaneous heating could be caused by improper and preventable conditions or by a wrong system of mining, whilst the second, stated that a mining engineer has to work a mine safely and profitability as possible. He may then have to leave coal underground at the risk of spontaneous fires or to adopt methods of working that entail risk of heating.

2.2. GEOLOGICAL FACTOR:

2.2.1. Seam thickness: Where the seam thickness is greater than that which can be completely mined in one part, the area is more susceptible to spontaneous combustion, since the un mined area tends to be subjected to sluggish ventilation flow. It was found that spontaneous combustion was apparently dependent upon the physical factors involved by the thickness of the seam, the methods of working type of ventilation and the friable nature of the coal. Also, in thick seams certain bands within the section can be more liable to spontaneous combustion than others. The thicker the seam, the more difficult it becomes to avoid leaving relatively high risk coal within the goaf area. In some cases it is necessary to mine the waste. However, leaving the low risk coal in the waste invariably tends to be practically impossible. In certain cases, a coal roof and floor way thus be left where,

a) The natural floor or roof tends to be weak
b) The seam is thick, or

c) There are inferior coals over or below the seam.
d) There are inferior coals over or below the seam, in those cases there will undoubtedly be coal in a broken form places where it can heat, and the points of dander presented.

2.2.2. Seam gradient: Flat seams where bord & pillar and long wall methods are used are less susceptible to spontaneous heating. In an inclined seam control of combustion becomes more complex, since convection current resulting from the difference in the temperature must tend to cause air currents in the goaf. In addition, within the extracted areas, flow may be due to buoyancy as a result of the differing densities of the methane, carbon dioxide and nitrogen. Which again may influence the development of spontaneous combustion in the waste, goaf or old working?

2.2.3. Caving characteristics: In the mines where partial extraction is practiced, adequate pillars are left to support the superincumbent strata, and the caving characteristics are generally of little significance.

In order to reduce the amount of leakage air flow with in the extracted areas, it is desirable for waste to be filled with as fine a material as possible .Thus, this material occupies the greatest volume and fills the void. The type of friable waste and sufficient emphasis should aid on the non- combustible or non-carbonaceous material.

2.2.4. Faulting: Faulted group frequently has an influence on spontaneous combustion. Any grinding action along the fault plane, with the resulting formation of the fine coal, may leads to spontaneous heating .A fault generally slows down the rate of face advance to a safe minimum, with the attendant risk of heat development.

2.2.5. Coal outburst: Coal outbursts usually occur in the harder formation rather than in the softer one and lower coals are more susceptible to spontaneous combustion. However, great care must be taken where there is a possibility of coal outbursts and spontaneous combustion occurring concurrently, as the danger of the products of an outburst i.e. finely powdered coal and /or methane, passing over the site of an active heating are very great.

2.2.6. Coal friability: The more friable the coal is the larger the surface area exposed to oxidation, thus tending to yield more heat per unit volume of coal.

2.2.7. Depth of cover: The depth of cover does not necessarily affects the risk of spontaneous combustion. In general the greater the depth of cover, the higher the natural strata temperature and thus the higher the base temperature of insight coal. With increase in depth strata temperature increases with a rate of 40m per degree centigrade.

Geothermal gradient does not directly affect the heating hazards, however, where geothermal gradient are high, the strata temperature in the working is likely to increase more rapidly with increasing cover than where the gradient is low.

2.3. MINING FACTORS

2.3.1. Mining methods: An advancing method of mining on the long wall system leaves extracted areas lying between the entries serving the working places .the ventilating pressure differences will encourage air to flow across these areas, with the accompanying risk of combustion. In high-risk situation a retreat system of working is normally preferred. However, the most important advantage gained by adopting a retreating system is lost if an attempt is made to ventilate the waste using a bleeder entry system.

2.3.2. Rate of advance: Within any waste area adjacent to working place there will be air entering the waste, either by the force of ventilating current or deliberately induced by a bleed action. in this area the rate of flow can be critical. In practice, when a working face is operating normally, any individual piece of coal passes through the zone at a rate equal to the rate of advance of the working place. It is the time taken from entering or leaving the zone that is critical. if the time is excessive, the oxidation may occur to an unacceptable degree and a glob fire could result.

2.3.3. Pillar Size: Pillar size has a direct influence on the liability to heat. Ideally, pillars should be of a size to avoid crushing. This size depends on the strength of the coal, the coal, the depth of the cover and the influence of other workings with in the vicinity. Increase methane emission is an indication of crushing, around pillars that subsequently spontaneously heated. Generally in coalmines, depending upon the depth of the seam from the surface, size of the pillar is determined. In bord and pillar working, at moderate depth if the pillar size is 30m *30m center to center depth then chances of crushing will be minimized.

2.3.4. Roof condition: Poor roof releases the shock waves to pass though easily and the development of crack increases, so poor roof conditions increases the liability to spontaneous combustion .these falls cavities, which have to be supported and are often filled with timber. Such areas are often sites of localized heating.

2.3.5. Crushing: Crushing is significant in two types of locations, at pillar and rib edges and at worked out areas, where a pillar is subjected to crush, a situation can develop where leakage paths are created, leading both to the flow of air into the coal and in some circumstances, through the solid to affect a more distant zone. Loose coal is usually present

in worked out areas, and is produced either by pillar spelling or by roof collapses with associated sluggish ventilation.

2.3.6. Packing: Where, packs are used, in seams, are liable to spontaneous heating. Experiences have shown that they must be of the highest possible quality. It was largely as a result of improvements in gate side packing the incidence of spontaneous combustion is generally reduced in most of the heading in a very short time.

2.3.7. Effect of timber: The timber props left in the waste caused the coal roof to disintegrate and created a saving thus encouraging the spontaneous heating of coal.

2.3.8. Road ways: Road ways in the seam liable to spontaneous heating are areas of concern due to leakage through crushed solid coal. The risk increases with thickness of coal, as this increases the area of coal exposed. The most common points for incidents are. Junction, air crossing, doors, regulators, connecting roads, obstruction in the roads, old roadways.

2.3.9. Leakage: To create the circumstances in which spontaneous combustion can occur, there must be a supply of oxygen and a situation where a built-up of heat is possible. This can be brought about by air leaks through fissures in solid coals and result in a shallow seated heating .this situation can occur where leakage paths exits at air crossings, in and around regulators and doors, and other similar locations where leakage there is a high pressure gradient and tendency for air to attempt to flow through solid coals. The leakage through a stopping depends partly on the difference in the pressure between the two sides of the stopping. Nearly all materials available for making the stopping are more or less permeable than others, it is not feasible to reply on making stopping impermeable and if they were completely impermeable, a danger pressure of fire damp could soon accumulate inside them. It should be done in the mind that the leakage trough the porous material , such as that of a stopping or well stowed goof varies in direct proportion to the difference of pressure on the two sides and not in proportion to the square root of the pressure.

2.3.10. Multi seam working: Where a multi seam situation exits both during the first seam and of subsequent seam, condition can arise with spontaneous combustion hazards for the seam currently being worked, and any other seams above or below of it .for example, where a seam has been worked with another un worked seam underlying it, leakage path can be created in to the lower seam, with a consequent risk of heating.

2.3.11. Coal losses: Coal losses that are, leaving remnant coal worked out areas are a series heating hazards and most gob fires result from this factor. There is no normal mining system that can guarantee that remnant will never be left in a waste area most mining

systems result in a significant loss of coal. The resulting situation, in which the coal is frequently likely to be crushed, finely divided, and in a location where built-up is possible, must be considered Potential hazards.

2.3.12. Worked out areas: Worked out areas are a potential sources of heating where such areas area not stopped off, there likely to have interruption in the ventilation system as result of roof falls or flour lift, leading to detoriation in rib condition and the presence of loose, small crushed coal- all such factors contributing to a potential hazards.

2.3.13. Heat from machines: Normally heat from machine is dissipated with in the ventilating air –stream and the temp rise of the general body of the air is likely to be very small. In some circumstances, the effect of the heat from machines is secondary, in that additional air may have to be circulated and will require a higher ventilating pressure with consequent increase risk of leakage.

2.3.14. Stowing: Stowing is carried out in the no of coal fields around the world specifically in order to seal mined out areas completely. As such, it has proved to be an effective method of spontaneous control.

2.3.15. Ventilating pressure: The flow of air in the mines is necessarily associated with pressure difference. This pressure difference is created by the mine fans and the natural ventilation, whist the pressure distribution underground depends upon the resistance of the air ways and distribution of the quantity. Air will try to flow wherever there is a path and a pressure difference, and this means that air will flow along any break or crack in the strata that is open to a pressure difference. A high ventilating pressure differential appears to be an important factor contributing to the spontaneous heating.

2.3.16. Barometric Pressure: It is generally agreed that air may find its way into a sealed-off areas as a result of at least one of the following causes

a) Continuous leakage, resulting from a difference in pressure on the return and intake stopping.
b) Barometric changes
c) Fluctuation in ventilating pressure resulting from the opening of doors and the movement of edges and mine cars.

2.3.17. Humidity: If coal absorbs moisture from the ventilating air it will heat up due to the release of the latent heat of condensation and chemisorptions effects. On the other hand, if the coal loses water by evaporation to the ventilating air, the reverse occurs if there is any imbalance between moisture in the air.

The balance between the complex mining condition that can bring about condensation or evaporation has not fully been researched but it is becoming increasing evident that changes in environmental moisture play a significant role in tipping the balance between adsorption and heating an evaporation and cooling. In Indian coal mines there were 17 cases of pillar fires between 5th september1965 and 8th March 1974.Out of these 17 cases of pillars fires,14 occurred during the dry season when the relative humidity remained low. At Hirapur colliery, there were & cases of fire in a5.6m seam.

2.4 THEORIES OF SPONTANEOUS COMBUSTION

2.4.1 Pyrite theory: Heating due to oxidation of pyrites has been known to be a common phenomenon in pyrite mines. Heating of coal can be caused by iron pyrites (only when present in considerable proportion) and in finely powdered and dispersed state in the presence of moisture. The reaction of iron pyrites with oxygen and moisture is exothermic yields products of greater volume than the original pyrite thus opening more pore area for oxygen. The reaction can be given as

$$Fe\ S_2 + 7 0_2 + 16 H_2O = 2\ H_2SO_4 + 2\ Fe\ S0_4.7\ H_2O + 316\ kcal$$

The oxidation of pyrite during weathering of coal seam may be represented by

$$Fe\ S_2 + 15/4\ O_2 + 9/2\ H_2O = Fe\ O.OH + 2\ H_2SO_4$$

The equations indicate that both oxygen and moisture, two prime weathering agents, contribute to pyrite alteration and the sulphuric acid is formed as a by-product of the alteration. In comparison to dry coals in the presence of moisture, the reactivity of coal is found to be double and if pyrite is in finely dispersed state, it becomes 10 fold. It was seen that pyrite bellow 5% showed negligible effect.

2.4.2 Bacterial theory: The contribution of heating due to the action of bacteria cannot be completely ruled out. Spontaneous heating observed in haystacks and in wood are known to be mainly due to bacterial action. Different evidences showed that bacteria were capable of living on coal and in some cases such bacteria caused a slight rise in temperature of the coal. Graham observed that sterilized coal oxidized at the same rate as the un sterilized coal and concluded that mechanism of oxidation did not include bacterial activity. Fuchs however concluded that bacteria could cause only a slight heating which may not play any significant role. There is no conclusive proof to authenticate or discard this theory.

2.4.3 Phenol theory: Experiments have shown that phenolic hydroxyls and poly phenols oxidize faster than many other groups. This theory is interesting because it offers a method of determining liability of coal to spontaneous heating.

2.4.4 Electro-chemical theory: It explains auto-oxidation of coals as oxidation-reduction processes In micro galvanic cells formed by the coal components.

2.4.5 Humidity theory: Quantity of heat liberated by atmospheric oxidation of coal is much less than the quantity of heat required removing water from the coal. Thus it can be concluded that if the evaporation of water can be induced at the seat of heating, then the temperature of heating would decrease. Mukherjee and Lahiri (1957) proposed the following mechanism of the reaction between water and coal at 100°C. (Brackets indicate chemisorptions):

H_2O - (H) (OH) -(H_2) (0) --(H_2) +0_2

C + 0 --- (CO) --- CO

(CO) + (0) --- (CO_2) --- CO_2

When it is recalled that water is an oxidation product of low temperature oxidation of coal, the above scheme well explains other possible sources of CO and CO_2 in low temperature reaction between coal and oxygen.

2.4.6 Coal-oxygen complex theory: Oxidation of coal is believed to be initiated at native radical sight. Formation of peroxyl radical and hydro peroxides is commonly to be thought to be they mechanism by which oxygen and moisture are initially in corporate into organic matrix These species may react, rearrange or decomposed to form wide range of oxygen functionality in the matrix or gaseous product.

Chapter 3

SAMPLING

CHANNEL SAMPLING PROCEDURE

SAMPLING

It is the process by which the physical and chemical properties of the mineral or ore can be ascertained with the desired accuracy.

It is the process of collecting the small portion of a whole such that consistence of that portion represents that of a whole.

Different types of sampling are:

- Channel sampling
- Chip sampling
- Grab sampling
- Bulk sampling
- Drill hole sampling
- Coning and quartering
- Rolling
- Splitting

Chip sampling is done in hard ores where it is difficult to cut the channels. It can be taken in case of uniform ores and where the rock structures are independent of the values. The sample is collected by breaking of small equal sized chips from a face at points usually equally spaced both vertically and horizontally.

Grab sampling is applied to the broken ore in the stope or at the face, ore transported. Usually grab sampling of the ore broken in the stope is unreliable as accurate estimation of the volume of broken ore is impossible. Grab sampling of tubs or ships is however more representations since samples are collected from units of regular volume.

Bulk sampling is done where conventional sampling methods do not give a representative scale; large scale sampling or bulk sampling resorted to. Bulk samples eliminate the effect of irregular distribution of value or minor.

3.1 CHANNEL SAMPLING PROCEDURE

Sampling was done by channel sampling method. This method consists of cutting channels across the face of exposed ore and collecting the resulting chips, fragments and dust from each channel to make up a sample.

Preparing the surface: Before cutting the sample the exposure has to be cleaned to remove dust, shine and soluble salts. This may be done by washing the rock thoroughly with a hose or scrubbing with a stiff brush. It is usually better to chip off the outer layer of the rock along the band within which the sample is to be taken.

When the surface is clean the next step is to mark out the location of the channel by inscribing to parallel lines on the rock a few inches apart, using as drawing material the smoke of a carbide lamp or else chalk or paint. The lines should be 3 to 6 inches apart, depending upon nature of rock and ore.

Cutting the channel: the tools are few but formidable a hammer and a moil. Instead of a hammer and moil a prospectors pick is convenient for very soft rock but it cannot cope with hard vein matter. In some operating mines the sampling process is speeded by using a light air operate machine drill equipped with a pointed or chisel shaped bit.

Collecting the sample: a sheet of canvas spread on the floor at the workings or on a platform will catch the chips as they fall, but unfortunately it may also catch dust and loosened blocks from outside the channel.

Labeling the channel: the collected sample is placed in a canvas sack along with an identifying label or tag. Some use numbered metal discs, others use block of soft wood on which a number is written with a hard pencil and protected with by covering the writing with another block and tying the two together.

Location of samples: since distribution of metals is apt to follow banding, a sample taken across it so as to include all of the bands is likely to be the most representative. Although a sample taken at right angles to the plane of layering will give the shortest length of cut, the channel may cross the layers at any convenient angle so long as it cuts all of the layers and cut them all at the same angles.

In drifts or steeply dipping veins the most convenient practice is to cut the samples across the back of the drift. In cross cuts the samples may be cut either horizontally or inclined perpendicular to the dip.

Subdividing sample channels: if an ore body has a width of much more than 5 ft. the convenient practice is to take two or more samples from each channel, sub dividing the channel in such a way that no individual sample represents a width of more than 5 ft.

Measuring sample widths: whether or not the samples have been cut normal to the walls of the vein the width recorded should ordinarily be the true width that is the width as measured along a line perpendicular to the vein walls. Measuring between projected planes of the walls or between projections of the points at which the channel has been sub divided usually adequate but if great accuracy is desired it is sometimes possible to make the projection by stretching a string from the vein wall, as exposed in the back of a drift.

Chapter 4

DIFFERENT THERMAL STUDIES FOR THE EVALUATION OF COAL OXIDATION TENDENCIES

ISOTHERMAL METHOD
ADIABATIC METHOD

CHEMICAL METHOD OR PUFF TEMPERATURE (PT) METHOD

TEMPERATURE DIFFERENTIAL METHOD

4. DIFFERENT THERMAL STUDIES FOR THE EVALUATION OF COAL OXIDATION TENDENCIES

Various methods have been used in laboratory for the study of relative tendency of coals to heat spontaneously. Most of the methods are based on the measurement of the oxidation rate and ignition temperature. The propensity of different coal to self heating has been studied in the laboratory using crossing point temperature, different thermal analysis, oxygen absorption method etc. the result of the test analyzed according to readily recognizable characteristics temperature and rates of heating under non adiabatic condition. These simple indices have historically been considered to be prediction of self heating liability.

The current research shows that crossing point temperature and those at which exothermically transforms from a relatively low level to more energetic one, fail to adequately identify known dangerous coal. The intensity of low level exothermically is more successful in this regard. The various methods may be broadly classified under the following heads:

1. Isothermal method
2. Adiabatic method
3. Chemical method
4. Temperature differential method

4.1 ISOTHERMAL METHOD

In this method coal sample is heated in a bath or furnace at constant temperature with oxygen or air passing through it at predetermined rate. The oxidation rate is determined which can be used as an indicator of coals potential for spontaneous heating.

4.2 ADIABATIC METHOD

The coal sample is placed in an insulated bath or oven chamber designed to maintain automatically the coal and its immediate surrounding medium at the same temperature so that no heat is lost or gained. During an experiment, the temperature of the sample is brought to a pre selected value (starting temperature) in a stream of flowing nitrogen and after the system has attained thermal equilibrium, the apparatus is put in to an automatic control mode, the nitrogen is shutoff and the sample is exposed to a flowing stream of preheated oxygen or air (dry or moist) when the temperature of the coal rises

above its surroundings. The temperature of the bath or the oven chamber automatically rises to coincide with the coal temperature. The experiment is run over a period of several hours up to 100 hours depending on the staring temperature and the oxygen concentration till an ignition occurs which is determined as the minimum temperature at which there is a steep rise in the temperature on the temperature–time curve. Coals may also characterized by their "adiabatic self heating time" which is the time interval between the staring of the experiment and ignition of the sample. By incorporating automatic oxygen feed regulating system, oxidation rate-adiabatic self heating time curves can be drawn.

4.3 CHEMICAL METHOD OR PUFF TEMPERATURE (PT) METHOD

The apparatus for the determination of "puff temperature" was based on Russian "chemical method" which consists of the following:

(1) A cylindrical copper block having 6 holes along the circumference and one at the center of the block. These holes have 15mm diameter and 200 mm depth. These 6 holes are for holding tight fitting reaction tube while the center one is for the temperature probe for measuring the temperature of the copper block. This design ensures the same rate of heating for all the reaction tubes.

(2) The reaction tubes are of 12 mm internal diameter and 250 mm in length, open at one end of which are connected to a horizontal manifold. One end of the manifold is provided with an aspirator bulb.

These six reaction tubes are filled with three samples, in duplicate, treated in the following manner.

Sample1 (original): 1 gm of sample is obtained from 750 mg of original coal sample (-40+60 mesh, Dp = 335 micron) with 250 mg of sodium nitrate mixed in a mortar.

Sample2 (fully oxidized): 1 gm of sample obtained from 750 mg of original coal sample (-40+60 mesh, Dp = 335 micron) with 0.5/cc perhydrol for 20 hours in darkness and then mixing with 250 mg of $NaNO_2$.

Sample 3 (fully reduced): 1 gm of sample obtained from 750 mg of original coal sample (-40+60 mesh, Dp = 335 micron) mixed with 4% by weight of benzedene and 250 mg of $NaNO_2$.

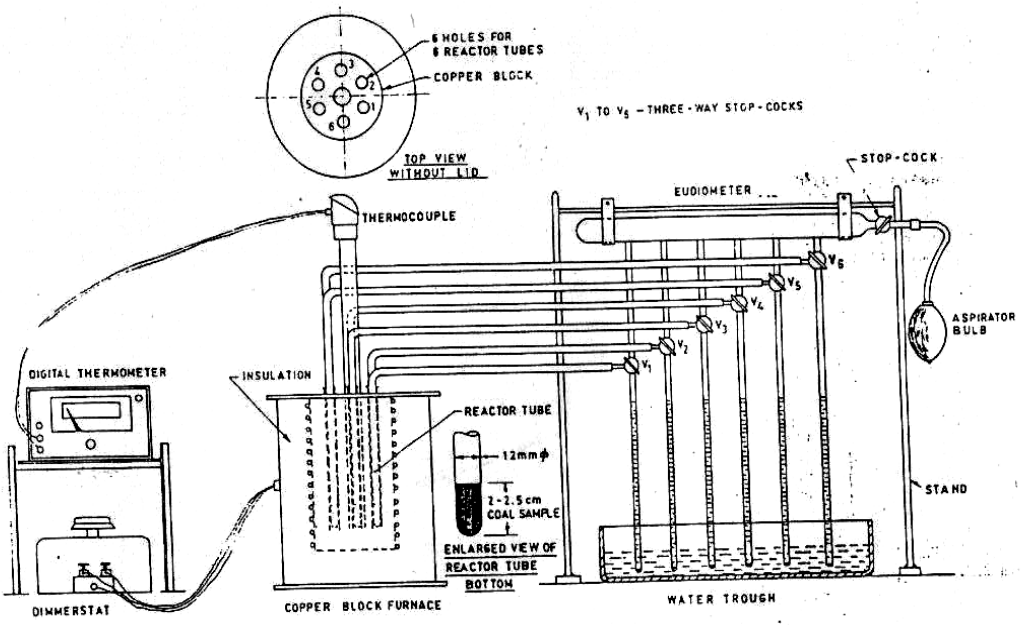

Fig 4.1 Schematic diagram of the apparatus for the determination of the ignition temperature of coal by chemical method

These reaction tubes were placed in to the holes of copper block. The water in the eudiometer tubes was raised up to a certain level by means of aspirator bulb in the eudiometer tubes are put on line with the reaction tubes. The furnace is switched on and the heating rate is adjusted to 4.5^0C/min. as the heating continued, the gases starts evolving from coal samples. When the coal temperature reaches a point, rapid evolution of gases occur this is indicated by the sudden fall of water columns in the eudiometer tubes. This temperature is noted which is known as puff temperature (relative ignition temperature). Depending upon the oxidation state of the coal samples, namely as received (T_p), fully oxidized (Tp_o) and fully reduced (Tp_r) ones.

The susceptibility potential and the degree of relative potential and the degree of relative oxidation are given as follows:

Susceptibility potential (SP) = (Tp_r-Tp)

Liability index (LI) = Tp_r-T

Relative degree of oxidation (RDOX) = ((Tp_r-Tp)/ (Tp_r-Tp_o))*100%

When, Tpr-Tpo= 10^0C, no danger of self heating

Tpr-Tpo<25^0C, less liable to self heating

Tpr-Tpo> 25^0C, very liable to self heating.

4.4 TEMPERATURE DIFFERENTIAL METHOD

The following methods are coming under this head

4.4.1 Crossing point temperature (CPT): The setup for the determination of crossing point temperature (CPT) of coal consists of following:

Vertical tubular furnace which has an internal diameter of 50mm and a heating capacity of 3kw. The furnace is provided with a temperature controller and a printer. Glass reaction tube is of 26mm internal diameter and 150mm in length. The reaction tube has spiraling glass tube of 6mm internal diameter around it which is connected to the bottom (inside) of the reaction tube for air inlet and a small out-let tube at the top acts as air/gas outlet. Flow meter and pressure flow control valves. Purifying and dehumidifying trains for air or nitrogen. A potassium hydroxide bubbler to remove carbon dioxide in the incoming air. A sulphuric acid bubbler to remove moisture in air. A drying tower containing granular calcium chlorides to remove moisture from air.

4gm sample of size -100+200 mesh (Dp = 112 micron) was placed in the reaction tube followed by glass wool at the bottom most position and a small sieve of 200 mesh (Dp = 72 micron) on that. The tube is then lightly tapped a fixed number of times to achieve uniform packing density of the samples. The reaction tube is then placed in the tubular furnace and a chromel-alumel thermocouple is inserted at the center of the sample. The entrapped air and occulted gases are removed from the coal samples by passing a mild current of nitrogen through the sample for three minutes, without disturbing the packing. The furnace is switched on and simultaneously air is allowed to pass through the sample, with an average rate heating of 10C per minute and at 80ml/ min. the temperature of the furnace (bath) and the coal sample are recorded at every five minute interval till the temperature of coal crossed over and gone beyond the furnace temperature.

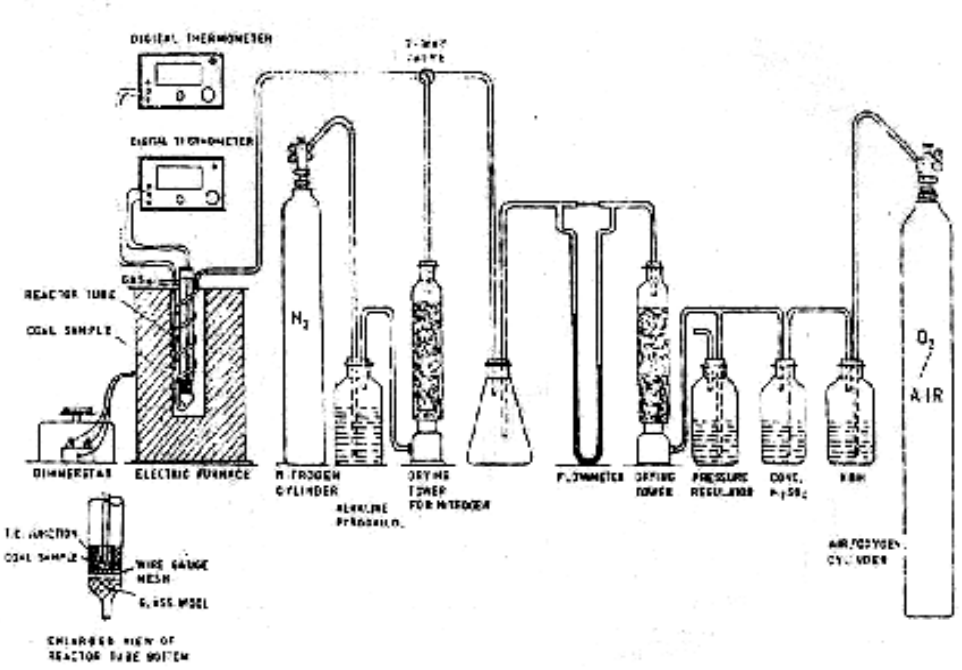

Fig 4.2 Schematic diagram of crossing point temperature

4.4.2 Flammability temperature method: The set up for the determination of the flammability temperature of coal consists of vertical tubular furnace of internal diameter 50mm, length 300mm, open at both ends, a dust dispersing unit, a solenoid valve a reservoir for air, a mercury manometer, a drying tower and an aspirator bulb. Coal dust sample is kept in the helical dust disperser. Air from the reservoir is made to pass through the disperser and on emergency from the divergent mount, forms a uniform dust-air mixture inside the furnace. The minimum temperature at which this mixture catches fire, which is indicated by the appearance of flame coming out of the bottom of the tubular furnace is the flammability temperature of the coal dust.

The furnace is switched on and the thermostat regulator is set at a temperature, near about the flammability temperature of known sample, by trial. 200 mg of coal dust of -200 mesh (Dp = 72 micron) size is put in to the dust dispersing unit. Keeping the solenoid valve and stop cock in closed position, air is pumped in to the reservoir through the drying tower using the aspirator bulb, until the pressure inside the reservoir reaches 8cm Hg. Stop cock (V1) is closed and stop cock (V2) is opened. Once the furnace reaches the pre-determined temperature, the solenoid valve is operated by cloud which passes through the tubular furnace. A sample is tried at various temperatures raising it from 200degree centigrade, in steps of 10 degree C. the temperature at which the flame

just appears, and is taken as the flammability temperature. The trials are made in triplicate and the mean is taken as the flammability temperature of the particular coal sample.

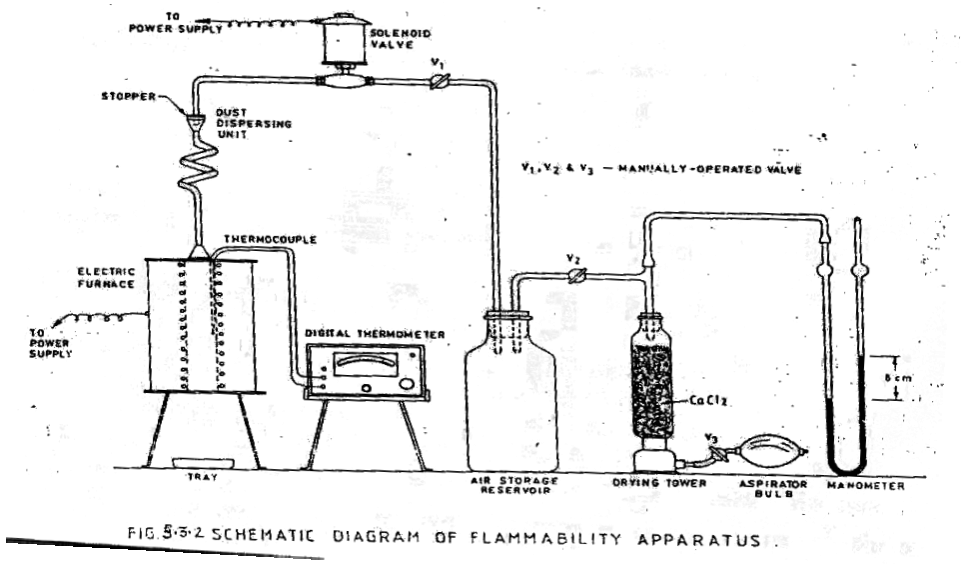

FIG. 5·3·2 SCHEMATIC DIAGRAM OF FLAMMABILITY APPARATUS

Fig 4.3 Schematic diagram of Flammability apparatus

4.4.3 Differential thermal analysis: DTA involves heating or cooling a test sample and an inert reference under identical conditions, while recording any temperature difference between the sample and reference. This differential temperature is then plotted against time, or against temperature. Changes in the sample which lead to the absorption or evolution of heat can be detected relative to the inert reference. Differential temperatures can also arise between two inert samples when their response to the applied heat treatment is not identical. DTA can therefore be used to study thermal properties and phase changes which do not lead to a change in enthalpy. The baseline of the DTA curve should then exhibit discontinuities at the transition temperatures and the slope of the curve at any point will depend on the micro structural constitution at that temperature.

A DTA curve can be used as a finger print for identification purposes, for example, in the study of clays where the structural similarity of different forms renders diffraction experiments difficult to interpret. The area under a DTA peak can be to the enthalpy change and is not affected by the heat capacity of the sample.

DTA may be defined formally as a technique for recording the difference in temperature between a substance and a reference material against either time or temperature as the

two specimens are subjected to identical temperature regimes in an environment heated or cooled at a controlled rate.

Experimental set up:

The key features of a differential thermal analysis kit are (Fig. 4.4):

1. Sample holder comprising thermocouples, sample containers and a ceramic or metallic block.
2. Furnace.
3. Temperature programmer.
4. Recording system.

The last three items come in a variety of commercially available forms and are not being discussed in any detail. The essential requirements of the furnace are that it should provide a stable and sufficiently large hot zone and must be able to respond rapidly to commands from the temperature programmer. A temperature programmer is essential in order to obtain constant heating rates. The recording system must have a low inertia to faithfully reproduce variations in the experimental set up.

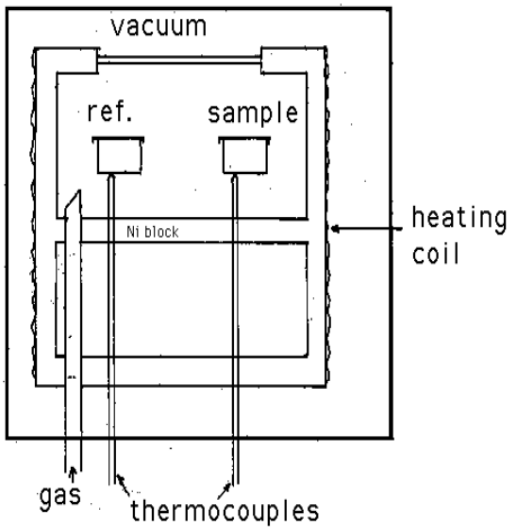

Fig. 4.4: Schematic Illustration of a DTA Cell

Fig. 4.5: Photographic view of SCHIMADZU DTA

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The sample holder assembly consists of a thermocouple each for the sample and reference, surrounded by a block to ensure an even heat distribution. The sample is contained in a small crucible designed with an indentation on the base to ensure a snug fit over the thermocouple bead. The crucible may be made of materials such as Pyrex, silica, nickel or platinum, depending on the temperature and nature of the tests involved. The

thermocouples should not be placed in direct contact with the sample to avoid contamination and degradation, although sensitivity may be compromised.

Metallic blocks are less prone to base line drift when compared with ceramics which contain porosity. On the other hand, their high thermal conductivity leads to smaller DTA peaks. The sample assembly is isolated against electrical interference from the furnace wiring with an earthed sheath, often made of platinum coated ceramic material. The sheath can also be used to contain the sample region within a controlled atmosphere or a vacuum. During experiments at temperatures in the range -200 to 500 ^{o}C, problems are encountered in transferring heat uniformly away from the specimen. These may be mitigated by using thermocouples in the form of flat discs to ensure optimum thermal contact with the now flat bottomed sample container, made of aluminum or platinum foil. To ensure reproducibility, it is then necessary to ensure that the thermocouple and container are consistently located with respect to each other.

Experimental Factors

Care is necessary in selecting the experimental parameters. For example, the effects of specimen environment, composition, size and surface to volume ratio all affect powder decomposition reactions, whereas these particular variables may not affect solid state phase changes. Experiments are frequently performed on powders so the resulting data may not be representative of bulk samples, where transformations may be controlled by the buildup of strain energy. The packing state of any powder sample becomes important in decomposition reactions and can lead to large variations between apparently identical samples. In some circumstances, the rate of heat evolution may be high enough to saturate the response capability of the measuring system; it is better than to dilute the test sample with inert material. For the measurement of phase transformation temperatures, it is advisable to ensure that the peak temperature does not vary with sample size.

The shape of a DTA peak does depend on sample weight and the heating rate used. Lowering the heating rate is roughly equivalent to reducing the sample weight; both lead to sharper peaks with improved resolution, although this is only useful if the signal to noise ratio is not compromised. The influence of heating rate on the peak shape and disposition can be used to advantage in the study of decomposition reactions, but for kinetic analysis it is important to minimize thermal gradients by reducing specimen size or heating rate.

Experimental Procedure

In the current study he standardized parameters suggested by Banerjee and Chakravorty (1967) are followed while performing the experiments. DTA thermograms are obtained at certain temperature at a heating rate of 5 ^{o} C per min. It has been shown by Banerjee and Chakravorty (1967) that a thermogram of coal can be divided into three segments or stages. These stages have also been identified in the thermograms generated from the results of the experiments as indicated in Figure 4.6.

In the initial stage of heating (stage I), the endothermic reaction predominates, probably due to the release of inherent moisture in coal. In the second stage (stage II), the exothermic reaction becomes significant, but the rate of heat release is not steady all through, as it changes with temperature. A steep rise in heat evolution is observed in the third stage (stage III).

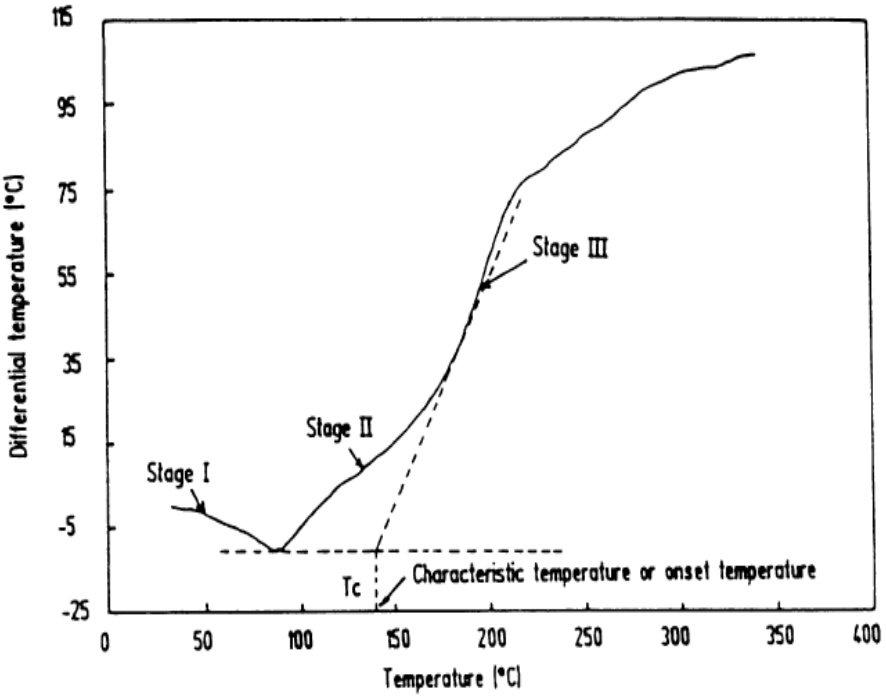

Figure 4.6 Different stages and characteristics temperature presented on the thermogram of DTA

The rate of temperature rise in stage II is cited by different researchers, viz. Banerjee and Chakravorty (1967), Gouws and Wade (1989); as being less for coals with less susceptibility to spontaneous heating. The exothermicity in stage III is not regarded as a reliable indicator of the self heating risk, because it may be equally high for low rank coals. However, the temperature of transition or characteristic temperature or onset temperature is considered to be significant. It is considered that in lower temperatures, the coal is more susceptible towards spontaneous heating.

Therefore, all the thermograms were analyzed for the following details:

Onset temperature or characteristic temperature

The onset temperature or characteristic temperature was determined by the following procedure:

1) A tangent was drawn at the inflexion point of the endothermic region and another tangent was drawn at the rising portion of the curve of stage III.

2) The intersection between the two tangents gives the characteristic temperature.

Determination of characteristic temperature or onset temperature for samples is determined by Figure 4.6.

Slopes of stage II of the thermogram

In the thermograms obtained from the experiments, linear stage II exothermicity is not observed. In view of the disjointed nature of stage II slopes it was further divided into two different regions, viz. stage IIA and stage IIB. The following three parameters of stage II were determined for further analysis.

(a) Average slope of stage IIA.

(b) Average slope of stage IIB.

(c) Overall slope of stage II.

Differential thermal analysis was carried out by a differential thermal analyzer. The standardized parameters suggested by Banarjee and Chakravorty (1967) were followed while performing the experiments. DTA thermo grams were obtained at 450^{o}C at a heating rate of 5^{o}C per minute on 60 mg of coal sample of size -212µ. Than the thermo grams obtained were analyzed in TA-60 software, using this software the peak of stage 1 is first calculated and then characteristic or onset temperature is identified at intersection point of the tangents at peak of stage 1 and at the starting point of stage 3. After determining the characteristic temperature than slope calculation at stage II A, II B and II were done using TA 60 software.

Thermo grams obtained after performing the DTA on samples for the 14 different coal samples are presented in Figure nos. 4.7 to 4.20. The onset temperature and slopes of the different stages obtained by following the above procedure is presented in table 4.3.

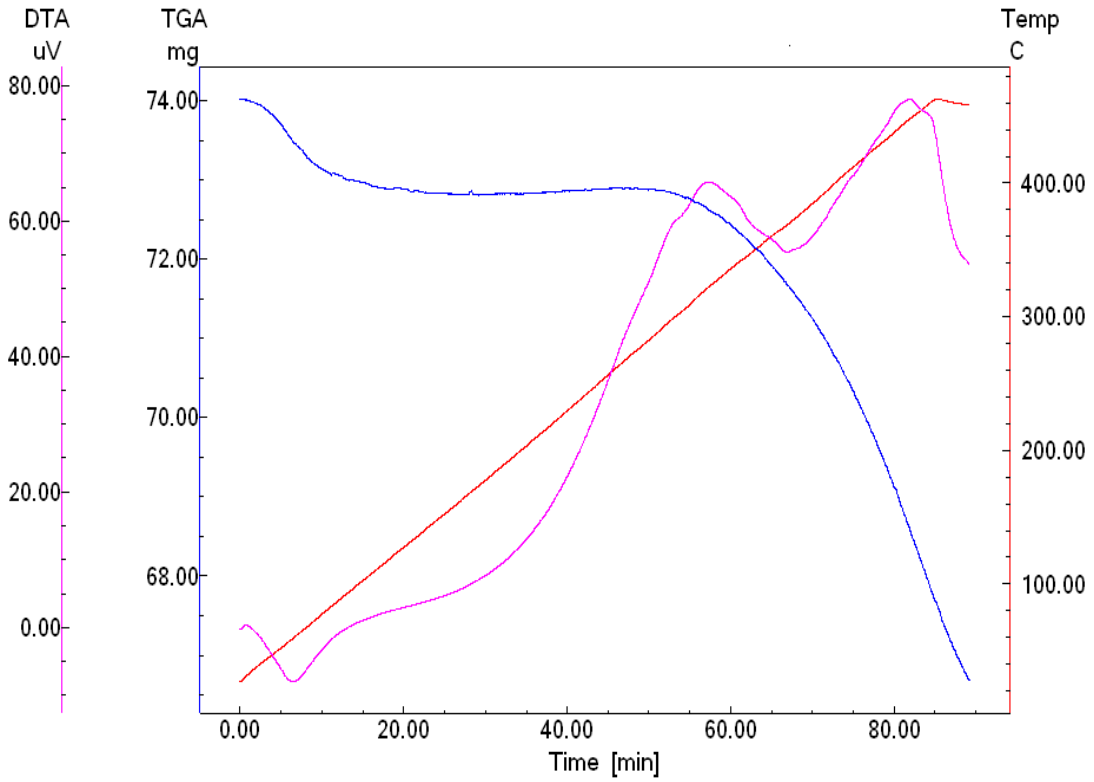

Fig 4.7: DTA Thermogram for sample no. 1

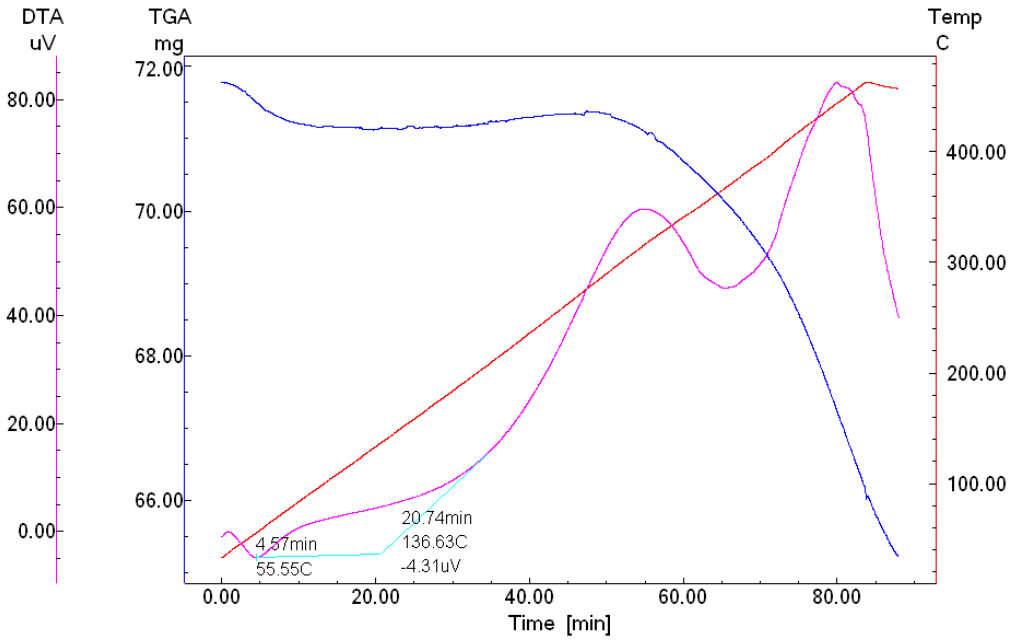

Fig 4.8: DTA Thermogram for sample no. 2

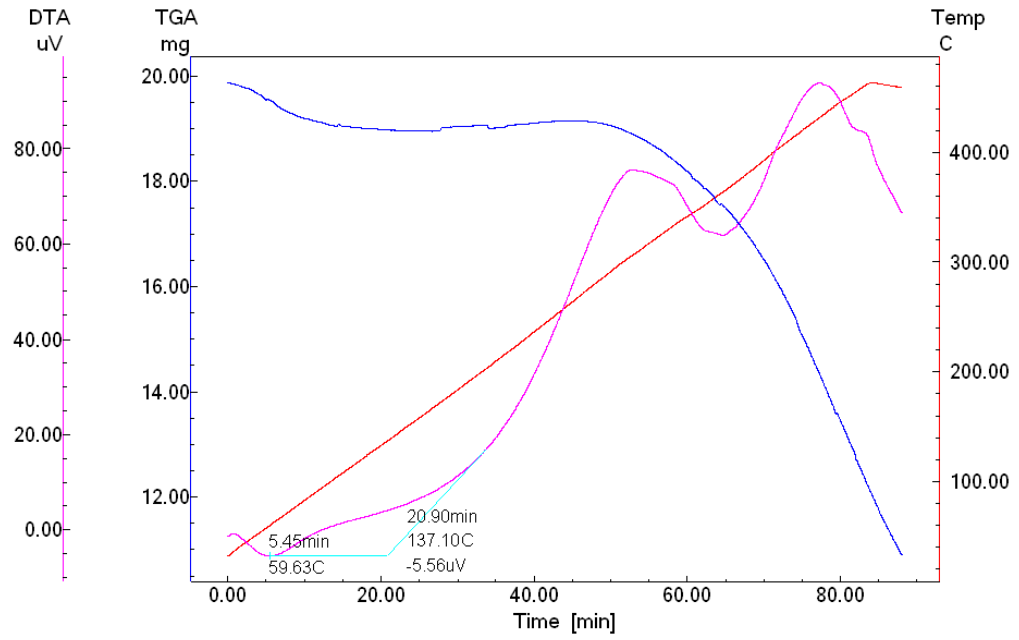

Fig 4.9: DTA Thermogram for sample no. 3

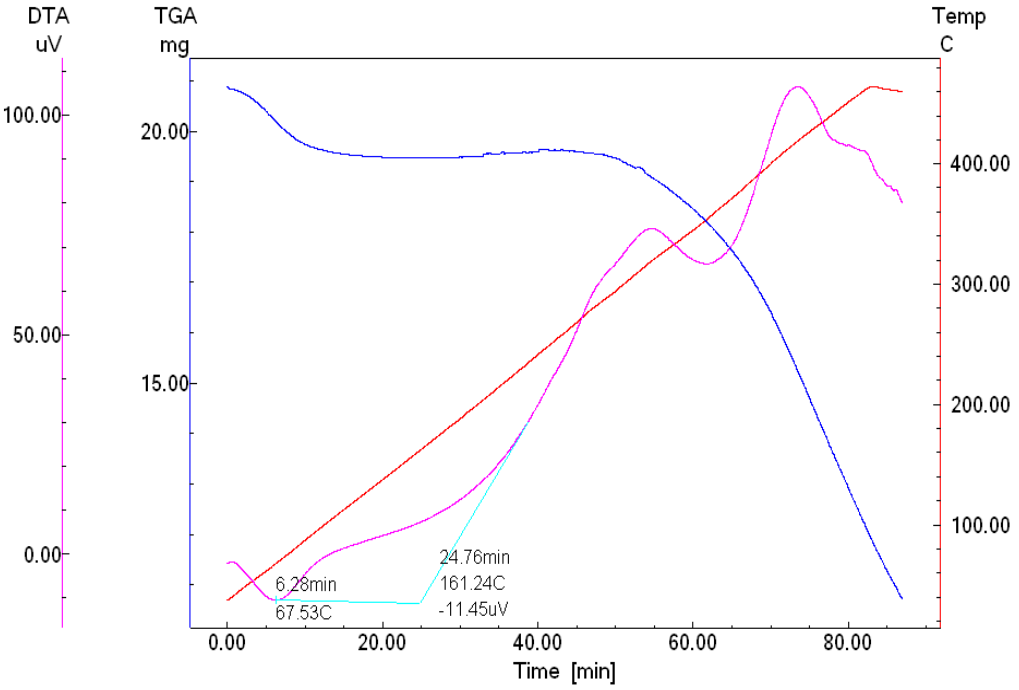

Fig 4.10: DTA Thermogram for sample no. 4

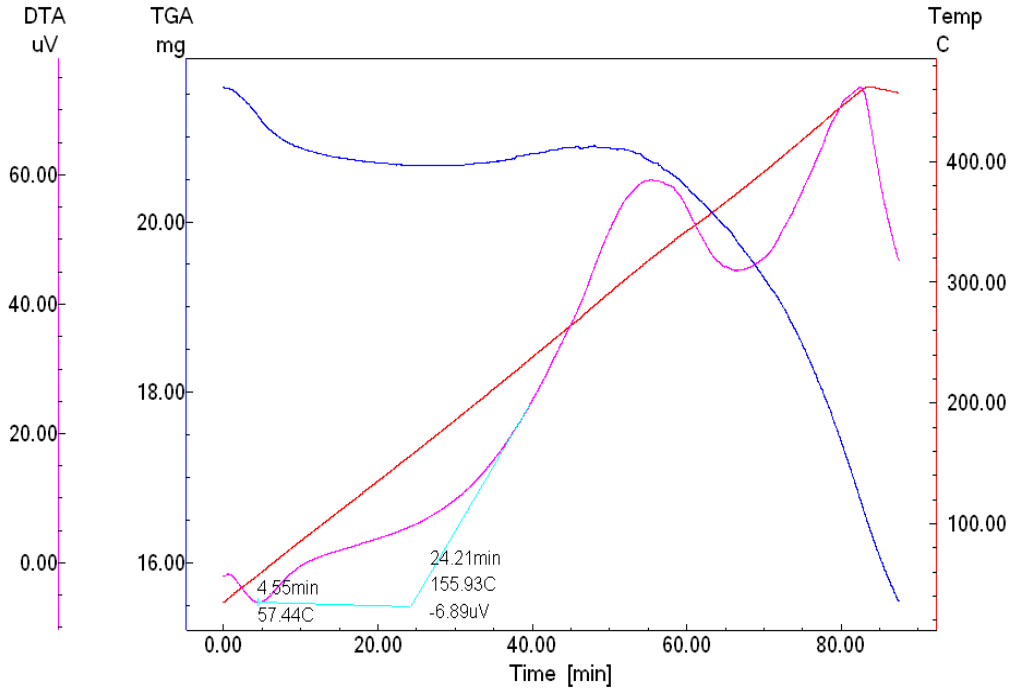

Fig 4.11: DTA Thermogram for sample no. 5

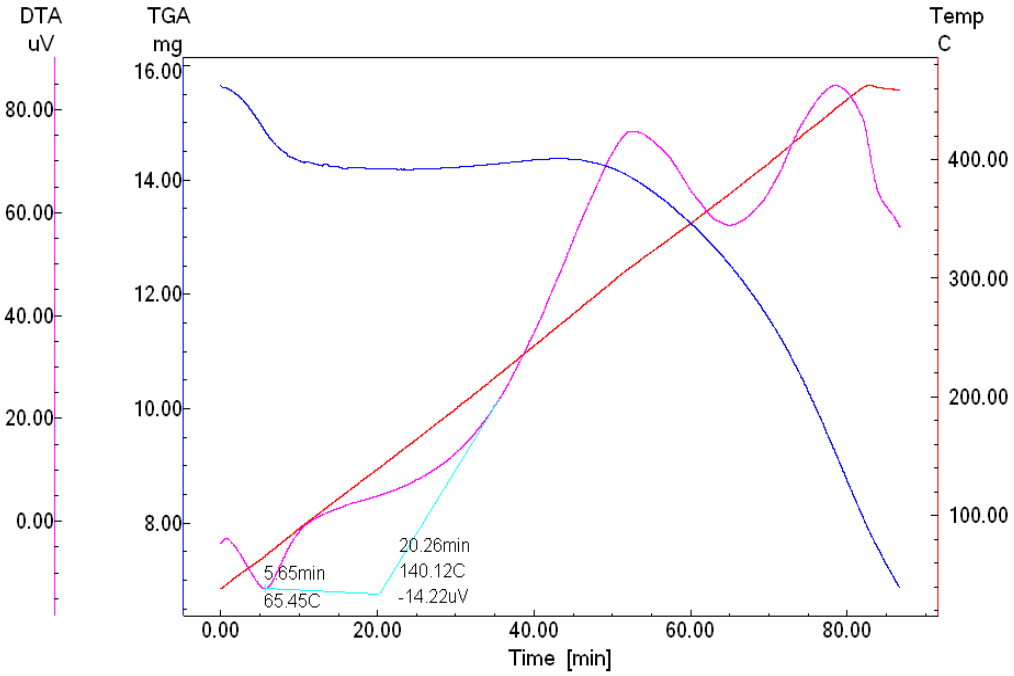

Fig 4.12: DTA Thermogram for sample no. 6

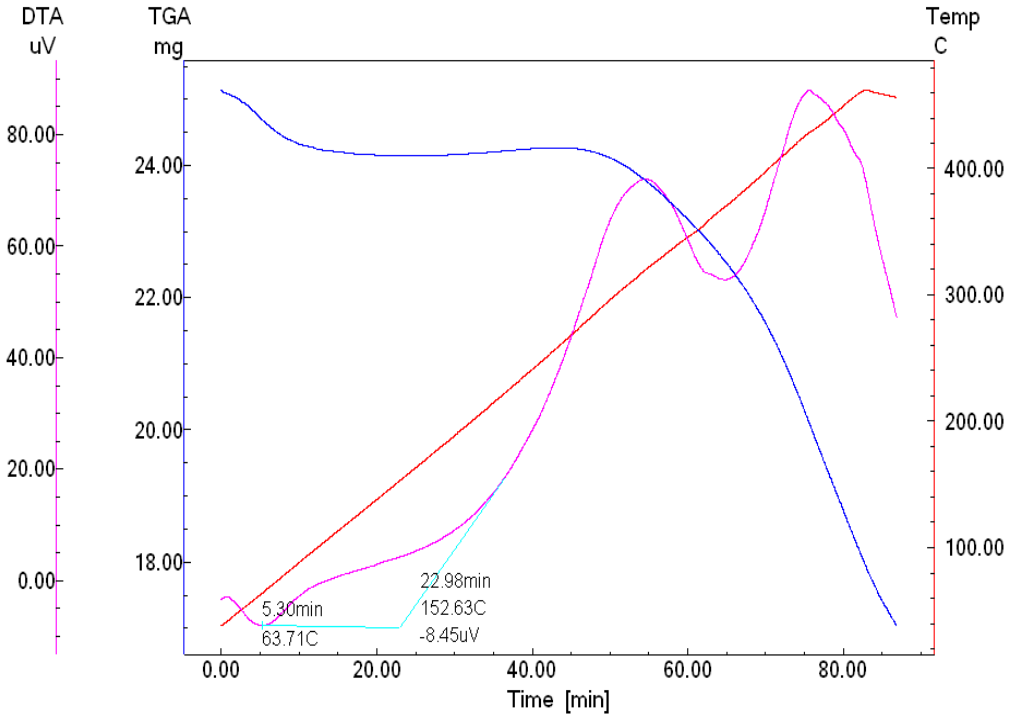

Fig 4.13: DTA Thermogram for sample no. 7

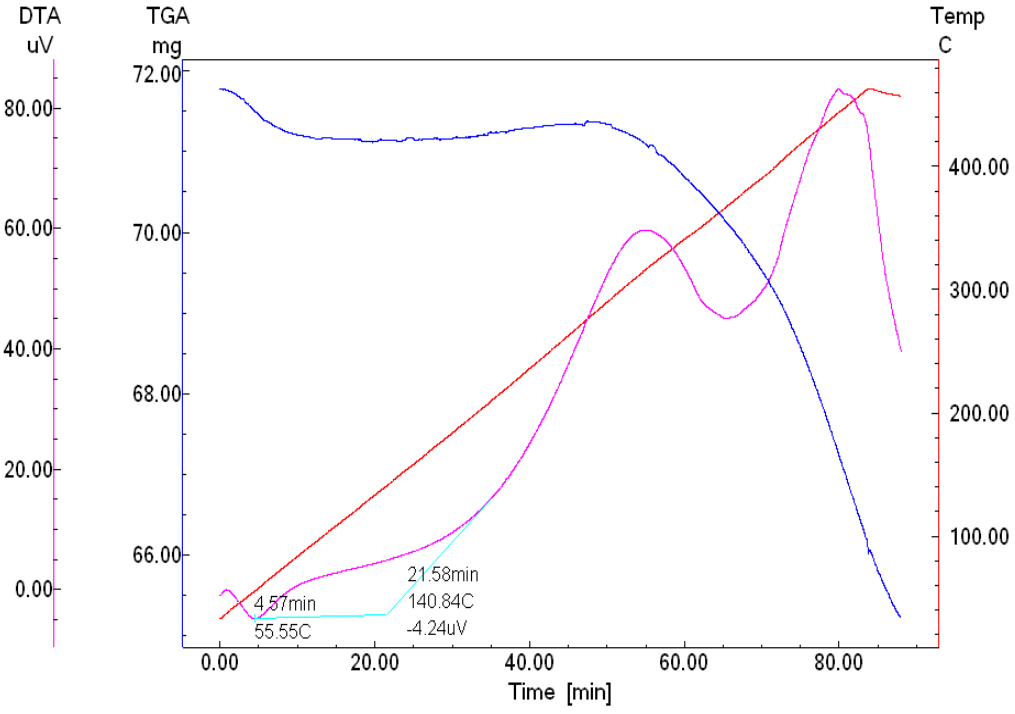

Fig 4.14: DTA Thermogram for sample no. 8

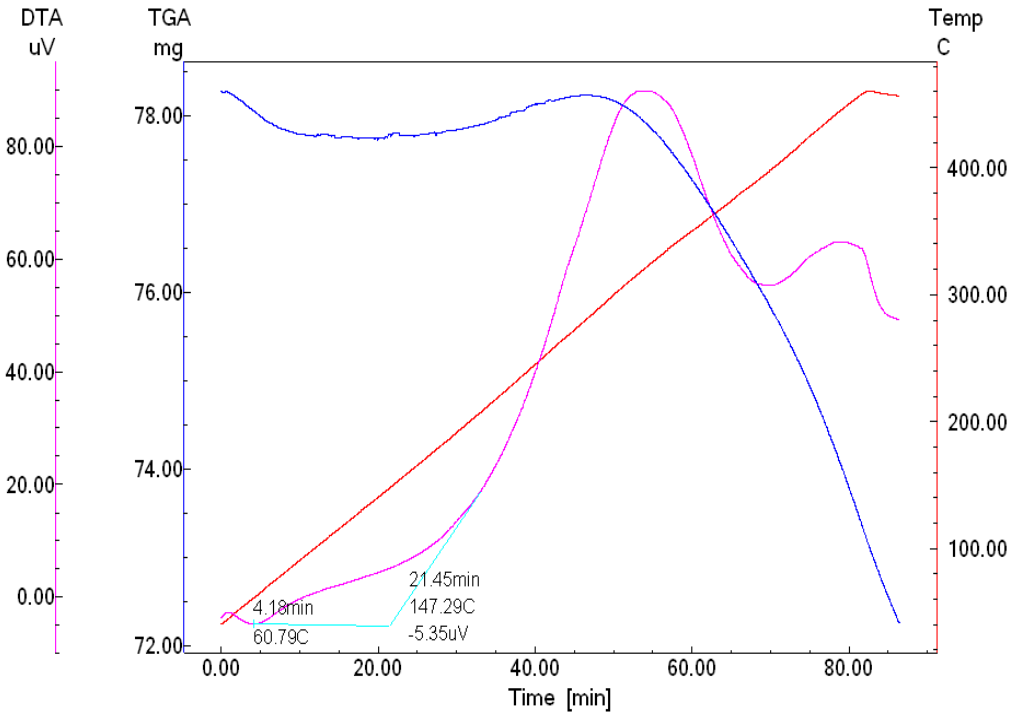

Fig 4.15: DTA Thermogram for sample no. 9

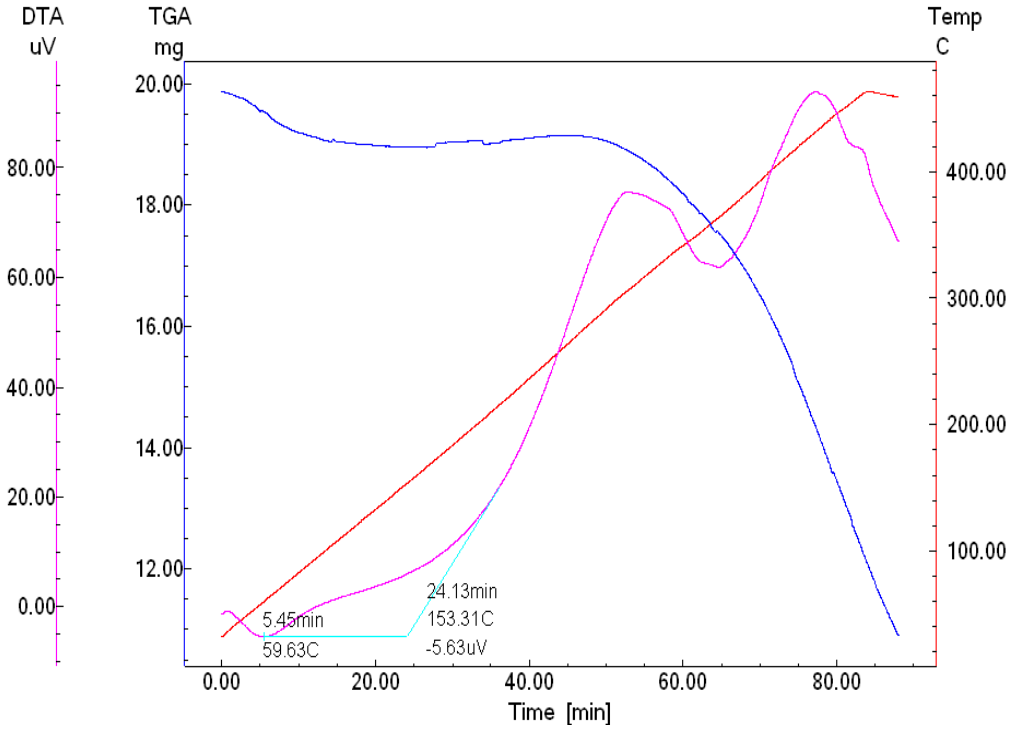

Fig 4.16: DTA Thermogram for sample no. 10

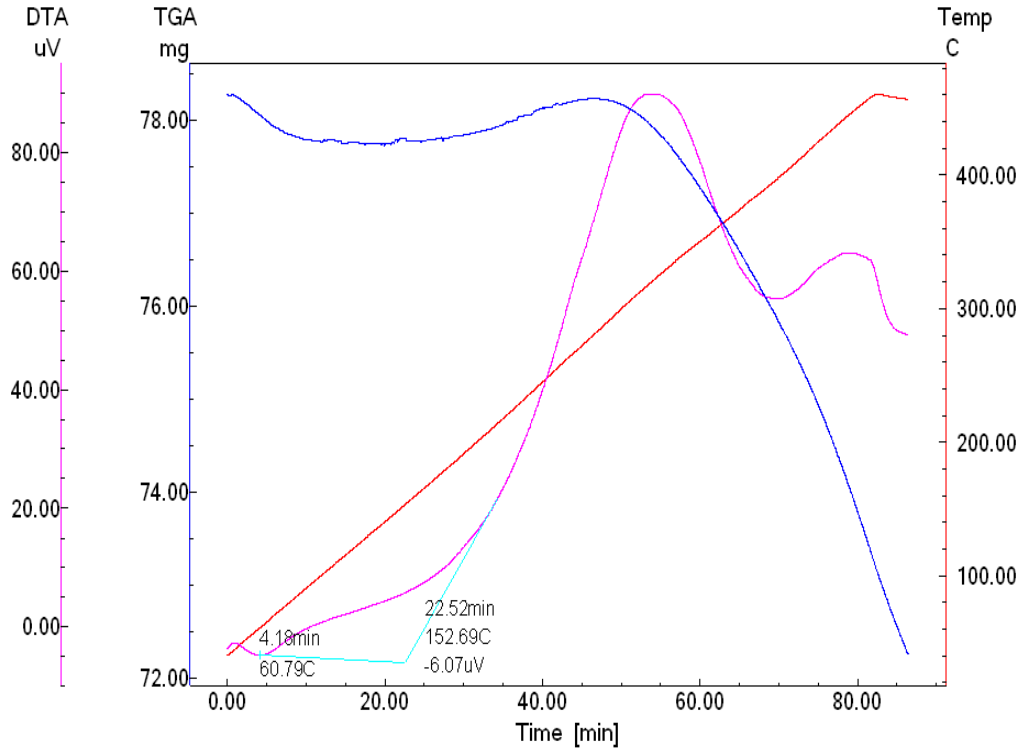

Fig 4.17: DTA Thermogram for sample no. 11

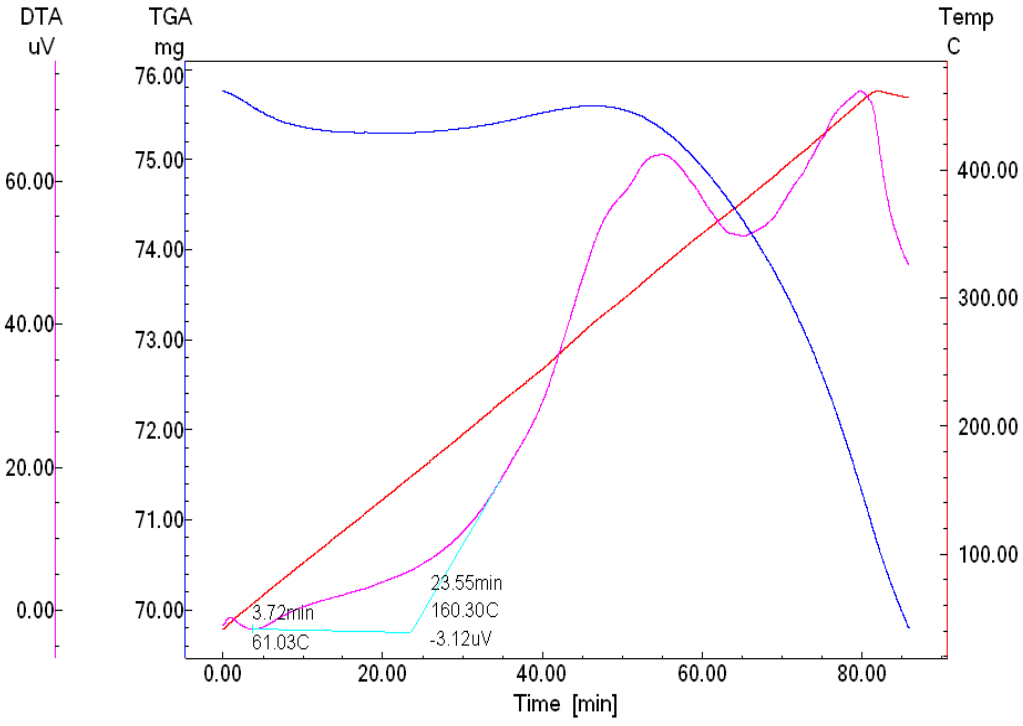

Fig 4.18: DTA Thermogram for sample no. 12

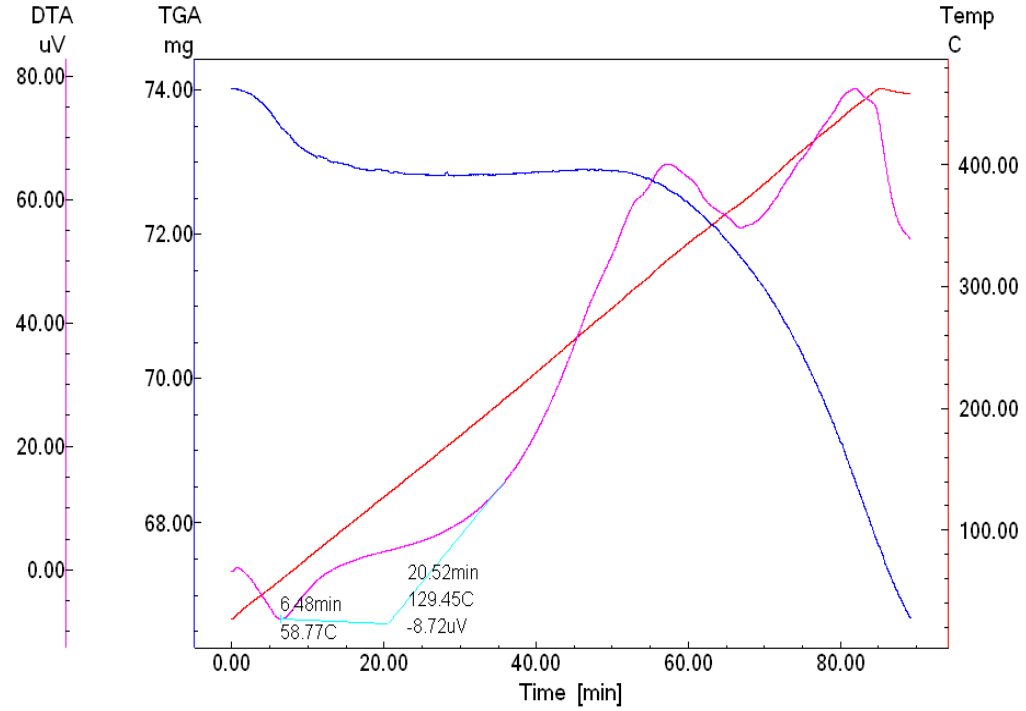

Fig 4.19: DTA Thermogram for sample no. 13

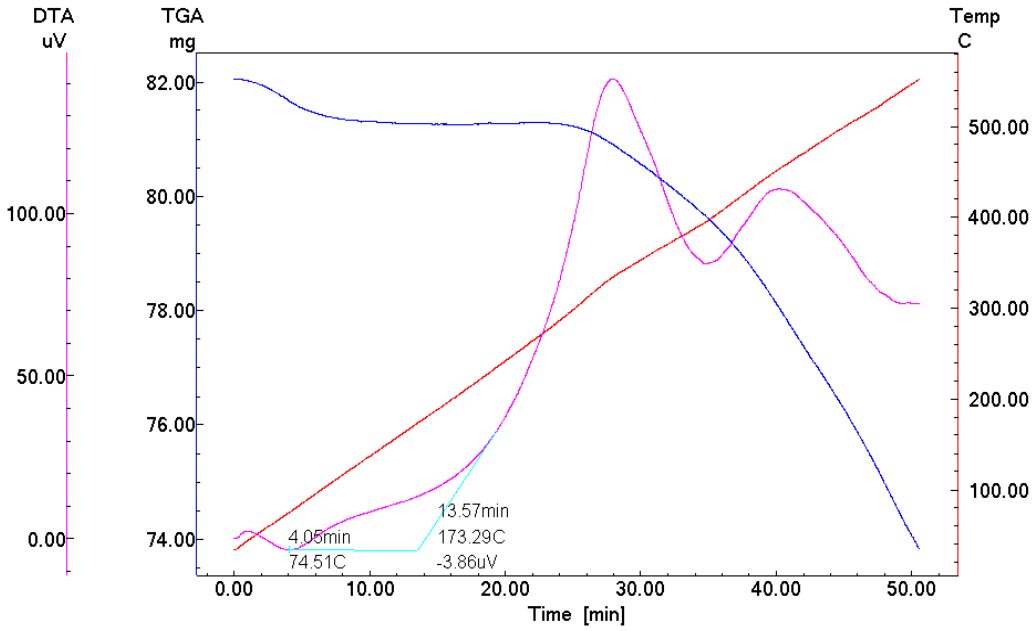

Fig 4.20: DTA Thermogram for sample no. 14

TABLE 4.3: Onset Temperature and Slopes for Coal Samples

Sample No.	Sample Name	Onset Temp. (in ^{0}C)	Slope Values		
			II A	II B	II
1	Mine 1	138.54	0.2258	0.1089	0.116
2	Mine 2	136.63	0.1628	0.1094	0.1028
3	Mine 3	137.10	0.1603	0.1426	0.1289
4	Mine 4	161.24	0.3006	0.1533	0.1617
5	Mine 5	155.93	0.1959	0.1138	0.1131
6	Mine 6	141.20	0.4149	0.1589	0.1674
7	Mine 7	152.63	0.1794	0.1751	0.1460
8	Mine 8	140.84	0.1942	0.1076	0.1035
9	Mine 9	147.29	0.1437	0.1536	0.1327
10	Mine 10	153.31	0.1540	0.1686	0.1439
11	Mine 11	152.69	0.1411	0.1483	0.1281
12	Mine 12	160.30	0.0923	0.1677	0.1318
13	Mine 13	129.45	0.3214	1.1056	0.5974
14	Mine 14	173.29	0.2061	0.1262	0.1198

Chapter 5

DISCUSSION AND RESULTS

DISCUSSION

CONCLUSION

5. DISCUSSION AND CONCLUSION

5.1 DISCUSSION

Coal mine fire is major concern for the Indian coal mining industry and the main cause of these fires is the spontaneous heating of coal. Apart from causing accidents, resulting in loss of lives and property, they also cause considerable economic losses to the organizations. Development of spontaneous heating in any section/part of a mine creates problems for the workings of other areas. Some of the other problems associated with spontaneous heating are diminution of heating values and coking properties, and in some cases serious environmental pollution. A careful analysis into occurrence of these fires reveals that most of these fires could have been averted if suitable preventive measures have been taken. It is therefore essential to first assess their degree of proneness in order to plan advance precautionary measures against the occurrence of these fires. In the present project work the spontaneous heating susceptibility of 14 coal samples have been assessed by differential thermal analysis experiments.

It may be observed from table 4.3 that the onset temperature of the coal samples varies from 129.45^0 (sample no. 13) to 173.29^0(sample no. 13). Lower is the transition temperature higher is the spontaneous heating susceptibility of coal seams. The IIA slope values vary from 0.0923 (sample no. 12) to 0.3214 (sample no.13), IIB slope varies from 0.1076 (sample no. 8) to 1.1056 (sample no 13) and overall slope of stage II varies from 0.1035 (sample no. 8) to 0.5974 (sample no. 13). It has been observed in the past that higher is the slope values higher is the spontaneous heating susceptibility of coal. Sample no. 13 has the lowest onset temperature and the highest slope values among all the samples. So it may be considered to be very highly susceptible. This sample belongs to the chirimiri coalfields and it is corroborated from the fact that it is known to be a fiery seam in the field. Panigrahi and Sahu (2004) have found that the coal seams having onset temperature in the range of 122^0C to 140^0C are highly susceptible to heating. Therefore, sample no. 1, 2, 3, 8 and 14 could be considered as very highly susceptible to heating.

They have also found that the seams having onset temperature between 140 to 170^0C to be moderately susceptible and above 170^0C to be poorly susceptible to spontaneous heating. Therefore, sample nos. 4, 5, 6, 7, 9 10, 11 and 2 can be considered as moderately susceptible and sample no. 13 as poorly susceptible to heating.

5.2 CONCLUSION

In India crossing point temperature is taken as the measure of spontaneous heating susceptibility of coals. However, it has been observed in the past that it does not predict the susceptibility for all types of coals, particularly for high moisture coals. Moreover, it takes approximately 3 hours to determine the CPT of a coal sample and failure of power supply for a few minutes during experiments, especially when the temperature approaches the crossing point, gives erroneous value of CPT and the whole experiment has to be repeated. In many cases the results are not reproducible.

Some of these demerits can be overcome by carrying out the differential thermal analysis, where it takes about 1.5 hour for the completion of an experiment. The results are highly reproducible and it is very easy to determine the transition temperature and slope values using the available thermal analysis software.

CHAPTER 6

REFERENCES

6. REFERENCES

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